Advancing Plasma-Based Technologies

PLASMIONIQUE

À l'Avant-Garde des Technologies Plasma

Your Partner in
Research and Innovation

FTIR Characterization of Carbon Nanotube Films - Coatings for the microbolometer.

Progress report 2013-04-01 to 2013-05-31

from ***Plasmionique Inc.***

Jean-Baptiste A. Kpetsu, ing., M.Sc.

Kazem Zandi, M.Sc.

Andranik Sarkissian, PhD

for ***DRDC Valcartier***

Suzanne Paradis, ing., M.Sc.

Philips Laou, PhD

Philippe Mérel, PhD

PBN: 14297 8295 RN0001

Tel: (450) 929 8154 Fax: (450) 929 8102

DRDC-RDDC-2014-C90

*Contract # **W7701-125241***

31 May 2013

CONTENTS

1. Growth and absorptance characterization of CNT films

In the last report, reflectance analyses performed on the old aligned carbon nanotube sample CS089N (CNT/Ni/TiN/Si) revealed a sharp drop in reflectance for the CNT coated sample as compared to its TiN coated Si substrate. New CNT samples are produced and analyzed hereafter for their transmittance, reflectance and absorptance in order to get a complete and more detailed picture of the near-to-mid IR absorbing properties of our CNTs.

1.1. New CNT samples

Three different materials namely titanium nitride (TiN), silicon nitride (Si_3N_4), and silicon dioxide (SiO_2) were used as sublayers on top of silicon substrates for the CNT growth. The samples with the electrically conductive TiN sublayer (CS122N) and the electrically insulating Si_3N_4 sublayer (CSiN-15N) led to CNT films that were suitable for the FTIR analyses. For the SiO_2 sublayer, both thermally grown commercial SiO_2 film (CSiOc01N) and sputtered SiO_2 coating (CSiO-07N) were used and none of them yielded a suitable CNT sample for FTIR analyses. It's worth mentioning however that for CSiO-07N, few CNTs spots were found on the sample after the CNT growth process but were removed from it by static electricity from a Petri dish cover. This is indicative of a very poor adhesion of the CNT layer to the SiO_2 sublayer. Therefore, it is very likely that CNTs were grown on our SiO_2 sublayer during the growth process but, due to their poor adhesion to the substrate, were almost completely swept away by gas flow or any other mean before the samples were removed from the PECVD reactor.
Detailed deposition parameters of all samples are available in the follow-up tables (A) and (B) of Annex 1.

1.2. Specular, diffuse, and total reflectance

Diffuse and total reflectance measurements were performed on samples CS122N and CSiN-15N using the integrating sphere accessory "A562-G/Q" with the FTIR spectrophotometer. The setup has been successfully modified to reduce the strength and the surface of mechanical contact between the CNT layers and the sphere. Thus, the samples are still of good quality and reliable subsequent SEM analyses could be performed to help correlate the optical and structural properties of the CNT films. Specular reflectance is calculated from the diffuse and total reflectance data and all the three spectra are shown in Figure 1 and Figure 2 respectively for CS122N and CSiN-15N. For comparison, specular reflectance spectra measured using the "A510/Q-T" accessory is also shown for each sample. The setup has also been modified for the latter accessory to avoid mechanical contact between its sample holder and the CNT layers. A plate from the heating stage unit "A599" has been used to fabricate a new custom sample holder adapted to the "A510/Q-T" accessory for fragile coatings such as CNTs, hence the added suffix A599-510 in the legend of the figures.

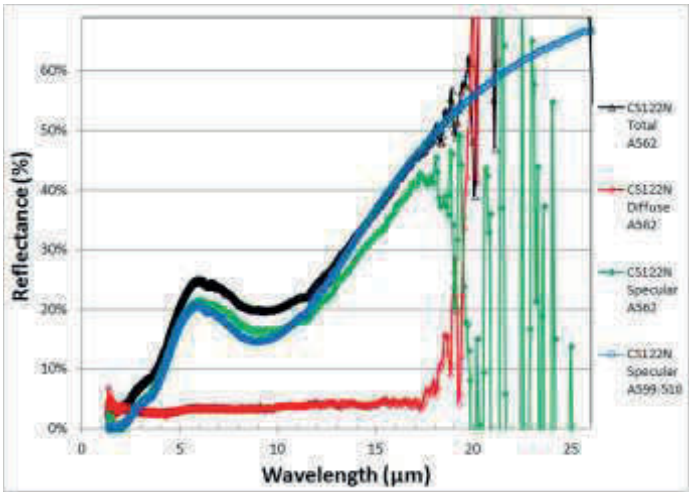


Figure 1 : Total, diffuse, and specular reflectance of a CNT/Ni/TiN/Si sample (CS122N) measured with the integrating sphere A562GQ and its specular reflectance measured with the A510QT accessory

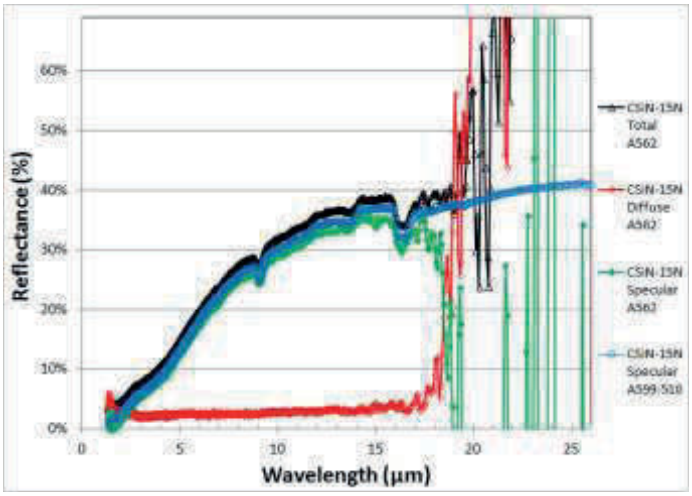


Figure 2 : Total, diffuse, and specular reflectance of a CNT/Ni/Si_3N_4/Si sample (CSiN-15N) measured with the integrating sphere A562GQ and its specular reflectance measured with the A510QT accessory

1.3. Transmittance and reflectance before and after CNT growth

Transmittance and reflectance measurements were performed on CNT samples CS122N and CSiN-15N as well as on their respective reference substrate S123N (Ni/TiN/Si) and SiN-16N (Ni/Si_3N_4/Si) using same successful procedures previously established for SiO_2 and Si_3N_4 samples [1]. Both types of measurement were performed using both the "A510/Q-T" and the "A562-G/Q" accessories with the same setup modifications described above. Especially for transmittance measurements with the A562GQ, the very low output signal limitation was overcome by using the high output power external MIR source mentioned in last report with an optimized combination of measurement parameters such as the aperture size, the signal gain, and the scanner velocity. The spectra obtained from the A510QT and A562GQ accessories are respectively shown in Figure 3 and Figure 4 for the pair (CS122N & S123N) and in Figure 5 and Figure 6 for the pair (CSiN-15N & SiN-16N).

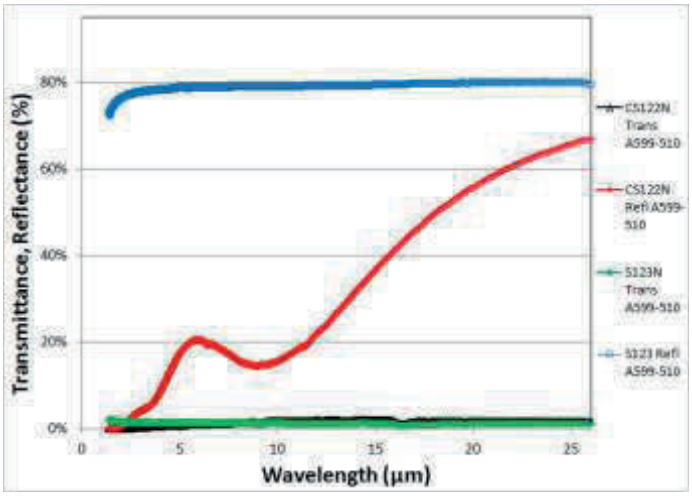


Figure 3 : Transmittance and specular reflectance of a CNT sample CS122N and its reference substrate S123N (Ni/TiN/Si) measured with the A510QT and its custom substrate holder from A599 plate

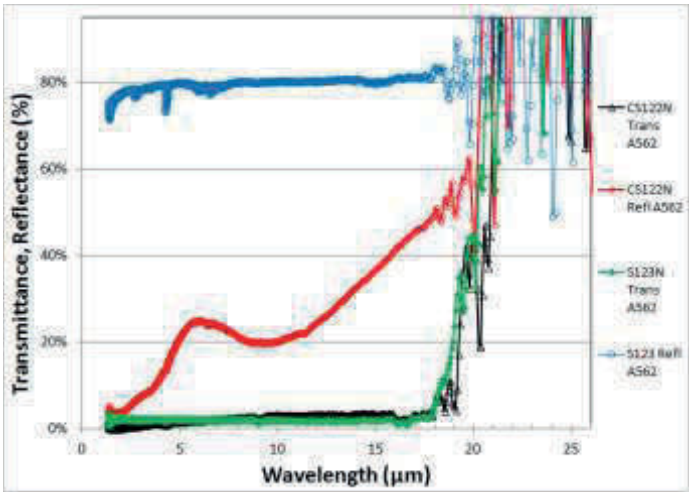


Figure 4 : Transmittance and total reflectance of a CNT sample CS122N and its reference substrate S123N (Ni/TiN/Si) measured with the integrating sphere A562GQ

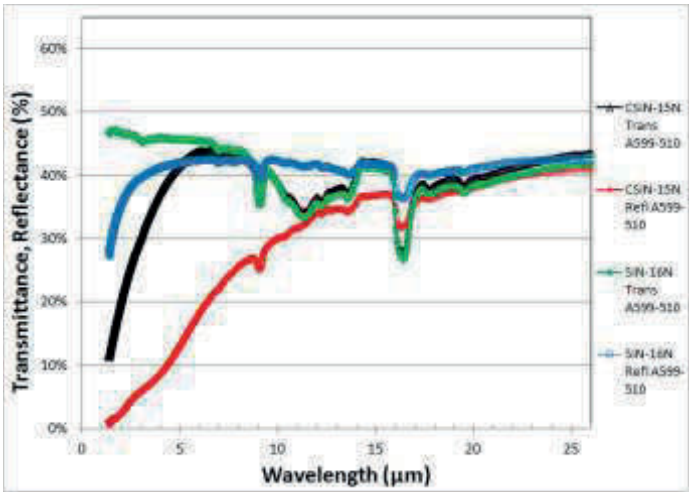


Figure 5 : Transmittance and specular reflectance of a CNT sample CSiN-15N and its reference substrate SiN-16N (Ni/Si_3N_4/Si) measured with the A510QT and its custom substrate holder from A599 plate

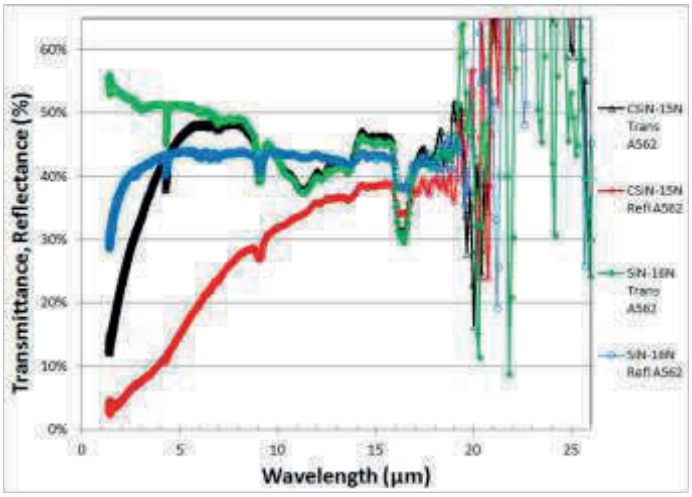


Figure 6 : Transmittance and total reflectance of a CNT sample CSiN-15N and its reference substrate SiN-16N ($Ni/Si_3N_4/Si$) measured with the integrating sphere A562GQ

1.4. Absorptance of the CNT layers

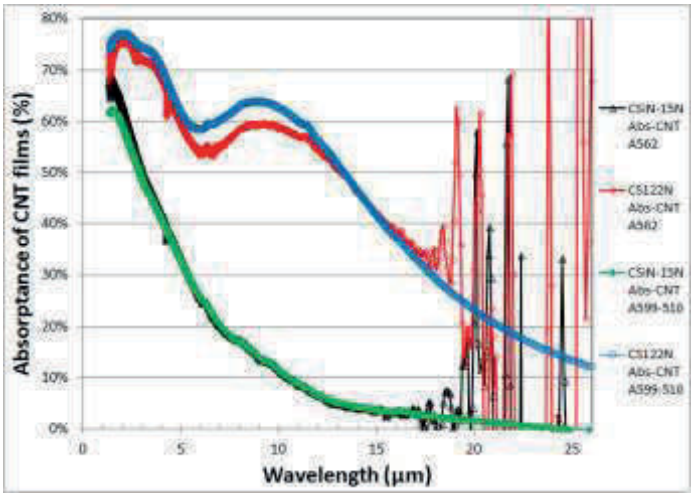


Figure 7 : Absorptance of the CNT layer of samples CSiN-15N and CS122N calculated from transmittance and reflectance data measured with the integrating sphere A562GQ and the A510QT accessory

The absorptance of the CNT layer of samples CS122N and CSiN-15N are calculated from the corresponding transmittance and reflectance data and shown in Figure 7. These results are quite different from the near-perfect black reported by Lehman *et al.* for their aligned CNTs of 162 µm of height [2]. Mizuno *et al.* also presented extremely low reflectance measurements of vertically aligned CNT arrays (less than 1-2%) indicating that it is possible to achieve extremely high absorptance at wavelengths ranging from the visible wavelengths to 200 µm [3].
Although both of our CNT films are still to be characterized by SEM, their height is not expected to be greater than 12.4 µm as previously measured for the sample CS089N (last report) which growth parameters were similar to CS122N. The quite lower absorptance of our films could therefore result to some extent from our much shorter CNT layer. The relatively high specular reflectance and low diffuse reflectance of our samples could be indicative of the CNT layer

density being so low that the almost completely specular reflection from the sublayer is predominant. The contrary could however also be true since it has been shown that for nanotubes that are close enough to touch, the coating's absorptance is about 75% while it rises to 99% when the CNTs are spaced a few tube diameters apart. [2] It is evidenced that the black body behavior is not an intrinsic nanotube property but stems from CNT coatings' unique forest structure, their sparseness and alignment [3].
The observed differences in absorptance for CS122N and CSiN-15N are thought to arise essentially from their different sublayer materials since their CNTs were grown simultaneously. Due to differences in their interfacial energy and interaction parameter, the different sublayer materials influence differently the nucleation and growth of the Ni catalyst layer leading to differences in the Ni layer properties (adhesion, particle size and distribution …) [4]. This in turn influences differently the CNT properties such as their diameter, density, growth mode (top or bottom), adhesion to substrate, and length [5].

2. Ti and YBCO coatings for the dual-band microbolometer

2.1. Sputter etching and new thick Ti deposition for posts

a. Sputter etching of residuals

SEM and EDS analyses performed on few VO_2 based bolometer prototypes that are being fabricated revealed some of the Ti posts were wiped out during lithography processes and were to be replaced as mentioned in last report. However, observed deposits at the bottom of some vias were suspected to consist of residuals, silicon nitride, and titanium. Concerns have been raised about the possible deleterious effects of these deposits and the native oxide on the electrical conductivity at the junction between the new Ti posts to be deposited and the gold (or chromium for some samples) layer underneath. To avoid such situation that could prevent any electrical measurement from being made with the prototypes, we proposed to use the plasma etching capability of the new Plasmionique SPT330 sputtering system to sputter clean the samples prior to the new Ti deposition. The sputter etch rates of the various materials were measured and the appropriate etching parameters determined for each sample based on the evaluation of thicknesses to be removed without damaging the underneath structure of the devices. Detailed etching parameters of all samples are available in the follow-up table (C) of Annex 1.

b. New thick Ti films deposition for posts

The need for a high deposition rate and good electrical properties led to the previously optimized Ti deposition parameters using the "CVC New-Sputter" equipped with a four-inch Ti target (second to last report). However, since the posts were swept away during lithography processes, posts with denser Ti were desired for the new films as it could reduce their etching rate and strengthen their adhesion to the underneath layer. Since the new Plasmionique SPT330 sputtering system was required for the above-mentioned sputter etching of residuals, the new recipe for denser Ti deposition was developed using the same system with a new three-inch Ti target. This was intended to prevent a new native oxide layer to form during the breaking of the vacuum for sample transfer between the sputter cleaning process and the new Ti deposition. Detailed deposition parameters for the new thick and denser Ti film are available in the follow-up table (C) of Annex 1.

2.2. Y-Ba-Cu-O films to be used as thermistors

New Y-Ba-Cu-O samples (YBCO-F to YBCO-J) were produced using the Plasmionique SPT330 sputter with deposition parameters chosen based on previous optimization results (last and second to last reports). The O_2 gas flow content in the Ar+O_2 gas mixture was kept at 10% in the 5-15% range previously determined and the working pressure was kept in a more conventional magnetron sputtering pressure range (2.5-15 mTorr) as compared to up to 150 mTorr previously for YBCO-A for example. Detailed deposition parameters for the YBCO samples are available in the follow-up table (C) of Annex 1. Film thickness and hence average growth rate were measured using the same lithographic and HCl wet etch techniques described in last reports. The resistivity and temperature measurement curves of the samples are shown in Annex 2. Their temperature coefficient of resistance (TCR) extracted from those data are available in Table 1 below.

Table 1 : Properties and few deposition parameters of various YBCO samples

Sample	Total Ar+O2 deposition pressure	O2/(Ar+O2) gas flow ratio	Thickness	Growth rate	Temperature coefficient of resistance (TCR)	Average resistivity at room temperature		
	(mTorr)		(nm)	(nm/min)	(%/K)	(Ω.cm)	±	±
Reported YBCO [6]					**-3.4%**			
YBCO-A	150	10%	116	0.6	-1.8%	1400	513	37%
YBCO-B	12	10%	252	2.0	-1.6%	2190	722	33%
YBCO-C	12	10%	210	1.7	-1.5%	614	155	25%
YBCO-D	42	10%	118	0.9	-1.5%	866	200	23%
YBCO-E	147	10%	47	0.4	-1.0%	646	289	45%
YBCO-F	5	10%	250	2.1	-1.5%	221	47	21%
YBCO-G	5	10%	198	1.7	-1.6%	1330	326	25%
YBCO-H	10	10%	138	1.2	-1.3%	883	200	23%
YBCO-I	15	10%	137	1.2	**0.0%**	1.5	0.41	28%
YBCO-J	2.5	10%	70	1.7	-0.7%	3.2	0.28	9%

Although the coatings of samples such as YBCO-A deposited at a relatively high sputtering pressure (150 mTorr) are supposed to have a composition much closer to a stoichiometric YBCO [1], their highest measured average TCR was -1.8%/K which is only ~53% of the reference reported value of -3.4%/K [6]. Moreover, YBCO-E, which was deposited at quite a similar pressure (147 mTorr) exhibited a quite different average TCR of -1.0%/K, which is even lower than the -1.6%/K obtained for YBCO-G that was deposited at 5 mTorr. It thus appears that the advantage of getting a coating with a composition closer to the stoichiometric YBCO by

increasing the sputtering working pressure do not relate in a simple manner to getting higher TCR. Reproducibility of the grown YBCO properties might also be a more important issue at higher pressures (YBCO-A and YBCO-E) than at lower pressure (YBCO-F and YBCO-G) as shown by the TCR data. Although a high TCR is the more important criteria for the use of our YBCO films as thermistors, it might be of good advice to use working pressures in the 5 to 15 mTorr range since our experiments show there's no guaranty of higher TCR at higher pressures while deposition challenges increase with such high working pressures. Elsewhere, porous film structures obtained at higher working pressures would've become another issue for the lithography patterning needed for our devices [7]. Pressures lower than 5 mTorr are not advised since it has been shown that substrate (Si) contaminants could be incorporated inside the YBCO coatings when the resputtering effect of the sample is too strong [1], which is more likely to occur at those low working pressures.

For YBCO-I, both negative and positive TCR values in the -1.2%/K to +0.9%/K were obtained with a strange average value of 0%/K. These results as well as those of YBCO-J are however not to be considered seriously since the YBCO target has been found to exhibit cracks after those last two depositions. The fissured target has been replaced with a newer and similar target which is to be used for upcoming depositions.

Bibliography

[1] J-B.A. Kpetsu, K. Zandi, C. Côté, and A. Sarkissian. *Thin Films Characterization and Optimization for a Dual-Band Microbolometer*, DRDC CR 2012-065

[2] J. Lehman, A. Sanders, L. Hanssen, B. Wilthan, J. Zeng, and C. Jensen, "Very black infrared detector from vertically aligned carbon nanotubes and electric-field poling of lithium tantalate," Nano Lett. 10, 3261–3266 (2010)

[3] K. Mizuno, J. Ishii, H. Kishida, Y. Hayamizu, S. Yasuda, D. N. Futaba, M. Yumura, and K. Hata, "A black body absorber from vertically aligned single-walled carbon nanotubes," Proc. Natl. Acad. Sci. USA 106, 6044–6047 (2009)

[4] Course Related Instructional Material for "Interfacial Engineering : Vapor Deposition Processes module", by D. Fennell Evans and Robert J. Stokes

[5] J-B. Kpetsu, C. Coté, P. Jedrzejowski, and A. Sarkissian/ P. Mérel, P. Laou, S. Désilets, and S. Paradis/ H. Liu, and X. Sun, "*Influence of Ni Catalyst Layer and TiN Diffusion Barrier on Carbon Nanotube Growth Rate*, " Nanoscale Research Letters **5**, 3 (2010) 539-544

[6] S. A. Dayeh, D. P. Butler, and Z. Çelik-Butler. *Micromachined infrared bolometers on flexible polyimide substrates*, Sensors and Actuators A 118, 49-56 (2005)

[7] J R. L. Sandstrom, W. J. Gallagher, T. R. Dinger, R. H. Koch, R. B. Laibowitz, A. W. Kleinsasser, R. J. Gambino, B. Bumble, and M. F. Chisholm. *Reliable single target sputtering process for high-temperature superconducting films and devices*, Appl. Phys. Lett. 53, 444 (1988)

Annex 1: Samples characteristics and follow-up table (#7)

Nomenclature for samples in the following table:

Si-0X : Si substrate with identification number 0X (**SN0X** for old samples).
ALN01 : Bare Al_2O_3 substrate, number 01.

Ti-0X: Si substrate with Ti coating, number 0X.
Bolo9Ti: Ti coating, on a special substrate (Bolo9 is the name of a device prototype used as substrate).
SX01 : Si substrate with a thermally grown oxide layer at 1000 °C in O_2, number 01.
S012 : Si substrate with TiN coating, number 012.
AL07 : Al_2O_3 substrate with TiN coating, number 07.
SiN-0Y : Si_3N_4 film with identification number 0Y deposited on an identified substrate.
SiO-0Z : SiO_2 film with identification number 0Z deposited on an identified substrate.
VO2-XX : VO_x film with identification number XX deposited on an identified substrate.
YBCO-YY : Y-Ba-Cu-O film with identification number YY deposited on an identified substrate.

SX18N : SX18 with Ni coating.
S100N : S100 with Ni coating.
SN11N : SN11 (Si substrate) with Ni coating, (usually for Ni calibration).
ALN02N : ALN02 with Ni coating.

CS100N : CNT grown on S100N
CALN28N : CNT grown on ALN28N.

Ti-18_pe: Plasma etching of sample Ti-18 using the Plasmionique SPT330 sputter.

A. Sputter deposition with the "CVC New-Sputter"

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter (")	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
TiN	12/07/09	NS	1	2	DC-1	TiN	10	Holder	10	3	1.5E-07	3.6E-07			98.6		5	10	495	0.204	50	0	0	1.23	3	3.7	2.1			4h00 pumping - Test before New CNTs
Ni		NS	8	4	RF-1	Ni	10	Holder	45	3					100.4	4.3	5	10	-810		0	0	30	0.75	1.7	1.3	1.55			Test before New CNTs
S118	12/07/10	NS	1	2	DC-1	TiN	10	Si	10	3	1.5E-07	3.4E-07		500	98.8		30	30	486	0.205	50	0	0	1.2	2.6	3.6	2.17		***100***	3h00 pumping - Heating: 25V,25A - For CNTs
S119								-						500	98.8		30	30	486	0.205	50	0	0	1.2	2.6	3.6	2.17		***100***	3h00 pumping - Heating: 25V,25A - For CNTs
S120								-						500	98.8		30	30	486	0.205	50	0	0	1.2	2.6	3.6	2.17		***100***	3h00 pumping - Heating: 25V,25A - For CNTs
S121								-						500	98.8		30	30	486	0.205	50	0	0	1.2	2.6	3.6	2.17		***100***	3h00 pumping - Heating: 25V,25A - For CNTs
Ni	12/07/18	NS	8	4	RF-1	Ni	10	Holder	45	3	1.9E-07	4.4E-07			100.4	5.5	30	0	-766		0	0	10	5.66	12	10	7.83			1h30 pumping, Cleaning
								-							100.4	4.3	0	30	-810		0	0	30	0.75	1.7	1.3	1.55			

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter ('')	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
Ni	12/07/19	**NS**	**8**	**4**	**RF-1**	**Ni**	10	Si	**45**	3	1.2E-07	2.8E-07			100.4	4.7	30	0	**-721**		0	0	10	5.76	13	10	7.88			1h30 pumping, Test with substrate
								-							100.4	4.3	10	2.5	**-807**		0	0	29	0.74	1.6	1.3	1.55		***2.78***	OK Coating observed
Ni	12/07/20	**NS**	**8**	**4**	**RF-1**	**Ni**	10	Si	**45**	3	2.3E-07	5.4E-07			100.4	3.8	10	0	**-733**		0	0	10	5.76	13	10	7.88			1h10 pumping, Test with substrate
								-							100.4	4.1	2	2	**-810**		0	0	30	0.74	1.4	1.3	1.55		***2.22***	OK Coating observed
Ni	12/07/23	**NS**	**8**	**4**	**RF-1**	**Ni**	10	Si	**45**	3	2.9E-07	6.7E-07			100.4	4.1	30	0	**-729**		0	0	10	5.58	12	10	7.88			0h40 pumping, Cleaning
S118N	12/07/23	**NS**	**8**	**4**	**RF-1**	**Ni**	10	S118	**45**	3	2.8E-07	5.9E-07			100.4	4.8	20	0	**-727**		0	0	10	5.72	13	10	7.9			1h15 pumping, Ni for CNT
S118N								S118							100.4	5.4	5	2.5	**-813**		0	0	30	0.76	1.4	1.3	1.56		***2.78***	
SN18N								Si							100.4	5.4	5	2.5	**-813**		0	0	30	0.76	1.4	1.3	1.56		***2.78***	
S119N	12/07/23	**NS**	**8**	**4**	**RF-1**	**Ni**	10	S119	**45**	3	3.0E-07	6.3E-07			100.4	5.5	15	0	**-725**		0	0	10	5.76	13	10	7.93			1h05 pumping, Ni for CNT
S119N								S119							100.4	4.3	5	3.75	**-816**		0	0	30	0.76	1.5	1.3	1.57		***4.17***	
SN19N								Si							100.4	4.3	5	3.75	**-816**		0	0	30	0.76	1.5	1.3	1.57		***4.17***	
S120N	12/07/23	**NS**	**8**	**4**	**RF-1**	**Ni**	10	S120	**45**	3	2.9E-07	6.0E-07			100.4	5.5	15	0	**-72**		0	0	10	5.79	13	10	7.95			1h10 pumping, Ni

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter (")	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																			8											for CNT
S120N								S120							100.4	4.3	5	5	-814		0	0	30	0.76	1.4	1.3	1.58		***5.56***	
SN20N								Si							100.4	4.3	5	5	-814		0	0	30	0.76	1.4	1.3	1.58		***5.56***	
SiTi-1	12/07/31	**NS**	**6**	**4**	**RF-1**	**Ti**	10	Si	**180**	3	1.1E-07	2.8E-07			100.4	3.8	2	0	-128		0		15	8.7	20	15	11.9			5h20 pumping + gas, Shutter closed
				4				-							200.9	0.8	2	3.6	-162		0		4.1	2.22	4.8	3.6	4.01	***24***		4 substrate passages in front of target for adhesion layer
SiTiAu-1			**2**	**2**	**RF-1**	**Au**	13	SiTi-1	**20**	3					50	1.8	5	10	-204		0		4.1	2.25	5	4	4.1	**168**		Gold layer for Mirror to be used for FTIR
Ni	12/10/09	**NS**	**8**	**4**	**RF-1**	**Ni**	10	holder	**45**	3	5.9E-07				100.4	5.4	5	0	-771		0	0	10	5.7	13		7.98			1h00 pumping after cleaning, Test
								-							100.4	4.1	5	0	-821		0	0	30	0.75	1.6		1.58			
Ni	12/10/09	**NS**	**7**	**2**	**RF-1**	**Ni**	10	holder	**45**	3					40.1			0												No plasma - high reflection not reduced
TiN	12/10/10	**NS**	**1**	**2**	**DC-1**	**TiN**	10	Holder	10													0	0							**Gas Alarm in the cleanroom, pumping all**

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter ('')	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																														night
TiN	12/10/11	**NS**	**1**	**2**	**DC-1**	**TiN**	10	Holder	10	3	1.4E-07	4.4E-07			98.6		0	25	**440**	**0.228**	50	0	30	2.02	4.7		4			Cleaning
								"							98.6		0	15	**475**	**0.21**	50	0	0	1.26	2.8		2.19			Cleaning
S122	12/10/11	**NS**	**1**	**2**	**DC-1**	**TiN**	10	Si	10	3	1.7E-07	3.7E-07		**500**	98.8		30	30	**482**	**0.208**	48.3	0	0	1.19	2.4	3.8	2.22		***100***	3h00 pumping - Heating: 25V,25A - For CNTs, DC Power and Voltage are -159W and 212V in the soft, different from MDX-1K controller values! The negative power prevents from automatic Wmin control of deposition. Chronometer used
S123								"						**500**	98.8		30	30	**482**	**0.208**	48.3	0	0	1.19	2.4	3.8	2.22		***100***	idem
S124								"						**500**	98.8		30	30	**482**	**0.208**	48.3	0	0	1.19	2.4	3.8	2.22		***100***	idem
S125								"						**500**	98.8		30	30	**482**	**0.208**	48.3	0	0	1.19	2.4	3.8	2.22		***100***	idem

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter ('')	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
SiO-07	12/10/12	**NS**	**8**	**4**	**RF-1**	**SiO2**	10	Si	**45**	3	2.1E-07	4.4E-07			400		50	0	**-642**			1	4	2.35	5.4	4.5	4.29			1h10 pumping
SiO-07			**8**	**4**				-							400		0	11.3	**-622**			1	4	2.08	4.7	4	3.78		***100***	Throttled - Software Fw/Reflected RF Power (476/39W) is very different from RFX-600 controller values (404/4W)! Automatic Wmin control of deposition from software values is still possible but not reliable. Chronometer used
SiO-08			**8**	**4**				-							400		0	11.3	**-622**			1	4	2.08	4.7	4	3.78		***100***	idem
SiO-09			**8**	**4**				-							400		0	11.3	**-622**			1	4	2.08	4.7	4	3.78		***100***	idem
SiO-10			**8**	**4**				-							400		0	11.3	**-622**			1	4	2.08	4.7	4	3.78		***100***	idem
SiN-15	12/10/12	**NS**	**8**	**4**	**RF-1**	**Si3N4**	10	Si	**45**	3	2.1E-07	4.7E-07		**200**	200		15	0	**-29**		1		7	4.37	10	8.5	6.45			2h00 pumping +

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter (")	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																			0												gas + 0h35 of 4x50 deg step-heating + 20min stabilization + Presputtering
SiN-15			**8**	**4**				-						**200**	300		20	8.84	**-365**		1		7	3.1	7.1	5.9	5.21		***100***	Deposition after 0h50 of heating. Throttled - Software Fw/Reflected RF Power (420/38W) is very different from RFX-600 controller values (301/2W)! Watt*Min-Done was 3125 at the end in software instead of the real 2651 for 8,84 min needed for 100 nm
SiN-16			**8**	**4**				-						**200**	300		20	8.84	**-365**		1		7	3.1	7.1	5.9	5.21		***100***	idem
SiN-17			**8**	**4**				-						**20**	300		20	8.8	**-**		1		7	3.1	7.1	5.9	5.2		***100***	idem

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter ('')	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
														0				4	365									1			
SiN-18			8	4				-						200	300		20	8.84	-365		1		7	3.1	7.1	5.9	5.21		*100*	idem
Ni	12/10/15	NS	8	4	RF-1	Ni	10	holder	45	3	3.8E-07	9.2E-07			100		0	75	-763		0	0	10	5.63	13	10	7.95			0h30 pumping for cleaning
S122N	12/10/15	NS	8	4	RF-1	Ni	10	S122	45	3	2.8E-07	6.1E-07			100		25	0	-725		0	0	10	5.73	13	10	8.02			1h40 pumping, Ni for CNT
S122N								S122							100		5	2.5	-810		0	0	30	0.76	1.5	1.3	1.59		*2.78*	
SiO-07N								SiO-07							100		5	2.5	-810		0	0	30	0.76	1.5	1.3	1.59		*2.78*	
SiN-15N	12/10/15	NS	8	4	RF-1	Ni	10	SiN-15	45	3	3.1E-07	6.3E-07			100		15	0	-720		0	0	10	5.79	13	10	8.01			1h15 pumping, Ni for CNT
SiN-15N								SiN-15							100		5	2.5	-810		0	0	30	0.76	1.7	1.3	1.6		*2.78*	
SiOc01N								SiOc01							100		5	2.5	-810		0	0	30	0.76	1.7	1.3	1.6		*2.78*	Commercial SiO2/Si/SiO2 substrate
Ni	12/10/16	NS	8	4	RF-1	Ni	10	holder	45	3	4.3E-07	9.3E-07			100		0	45	-778		0	0	10	5.68	13	10	7.97			0h35 pumping for cleaning
S123N	12/10/16	NS	8	4	RF-1	Ni	10	S123	45	3	3.0E-07	6.2E-07			100		15	0	-730		0	0	10	5.79	13	10	7.97			1h05 pumping, Ni for CNT
S123N								S123							100		5	2.5	-817		0	0	30	0.76	1.7	1.3	1.58		*2.78*	
SiO-08N								SiO-08							100		5	2.5	-81		0	0	30	0.76	1.7	1.3	1.58		*2.78*	

Sample name	Date	New-Sputter (NS) Plasmionique sputter (SPT)	Gun Number	Gun/Target diameter (")	Mode: DC or RF (Contactor/Generator 1 or 2)	Target	Target→Substrate Distance (cm)	Substrate (If in rotation)	Toggle Half-Angle (±X°)	Toggle Velocity	Base Pressure - Pfeiffer - Cold Cathode (Torr)	Base Pressure - Varian - Ion Gauge (Torr)	Real Temperature (°C)	Temperature Setpoint (°C)	Sputtering Power (W)	Reflected Power (W)	Presputtering Time (min)	Sputtering Time (min)	DC Bias in RF mode or Voltage in DC mode (V)	Current in DC mode (A)	N2 adjusted flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Varian - CDG (mTorr)	Deposition Total Pressure - Varian - ConvecTorr (mTorr)	Deposition Total Pressure - Pfeiffer - Pirani (mTorr)	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																			7												
SiN-16N	12/10/16	**NS**	**8**	**4**	**RF-1**	**Ni**	10	SiN-16	**45**	3	2.9E-07	5.8E-07			100		15	0	**-730**		0	0	10	5.81	13	10	7.99			1h15 pumping, Ni for CNT
SiN-16N								SiN-16							100		5	2.5	**-826**		0	0	30	0.77	1.8	1.3	1.59		***2.78***	
SiOc02N								SiOc02							100		5	2.5	**-821**		0	0	30	0.76	1.7	1.3	1.6		***2.78***	
S124N	12/10/16	**NS**	**4**	**2**	**RF-1**	**Ni**	10	S124	**10**	3	3.7E-07	7.6E-07			40		15	0	**-679**			0	28	16	37	28	23			1h00 pumping, Ni for CNT
S124N								S124							40		10	4.5	**-735**		0	0	28.5	0.73	1.5	1.4	1.51		***0!***	No coating (edge) observed! To be coated again
SiO-09N								SiO-09							40		10	4.5	**-735**		0	0	28.5	0.73	1.5	1.4	1.51		***0!***	idem
SiN-17N	12/10/16	**NS**	**4**	**2**	**RF-1**	**Ni**	10	SiN-17	**10**	3	4.0E-07	8.1E-07			40		15	0	**-676**			0	28.5	16.22	38	29	23.3			0h55 pumping, Ni for CNT
SiN-17N								SiN-17							40		5	4.5	**-740**		0	0	28.2	0.73	1.6	1.3	1.48		***7.1***	Coating is visible! Should have done 3min/4.3nm
SiOc03N								SiOc03							40		5	4.5	**-740**		0	0	28.2	0.73	1.6	1.3	1.48		***7.1***	Coating is visible! Should have done 3min/4.3nm

B. CNT growth with the PECVD system

Échantillons	Date	Pression de base - Jauge Pfeiffer (Torr)	Distance Substrat→Cible ou →Entrée µ-ondes (cm)	Substrat	Porte-substrats (PECVD)	Dépôt - Puissance (W)	Dépôt - Durée (min)	Dépôt - Température (°C)	Dépôt - Pression (mTorr)	Dépôt - Gaz H2 (%)	Dépôt - Gaz CH4 (%)	Dépôt - Gaz C2H2 (%)	Épaisseur (nm)	Polarisation DC (V)	Autres - commentaires (*Chiffre en italique = valeur estimée en fonction de mesures précédentes*)
CSN18N	12/10/17	2.2E-07	27	SN18N	BN	900	20	700	6000	80	20			0	CNTs delaminates, poor Ni adhesion
CSN19N	12/10/17	2.2E-07	27	SN19N	BN	900	20	700	6000	80	20			0	CNTs delaminates, poor Ni adhesion
CSN20N	12/10/17	2.2E-07	27	SN20N	BN	900	20	700	6000	80	20			0	CNTs delaminates, poor Ni adhesion
CSN21	12/10/17	2.2E-07	27	SN21	BN	900	20	700	6000	80	20			0	FTIR reference for 3 samples above, same Si - No Ni, no CNT
CS118N	12/10/18	1.4E-07	27	S118N	BN	900	20	700	6000	80	20			0	CNTs on Ni/TiN/Si
CS119N	12/10/18	1.4E-07	27	S119N	BN	900	20	700	6000	80	20			0	CNTs on Ni/TiN/Si
CS120N	12/10/18	1.4E-07	27	S120N	BN	900	20	700	6000	80	20			0	CNTs on Ni/TiN/Si
CS121	12/10/18	1.4E-07	27	S121	BN	900	20	700	6000	80	20			0	FTIR reference for 3 samples above but heating partially evaporates TiN - Avoid heating for next references
CS122N	12/12/14	1.8E-07	27	S122N	BN	900	20	700	6000	80	20			0	CNTs - for FTIR and SEM analyses
CSiN-15N	12/12/14	1.8E-07	27	SN19N	BN	900	20	700	6000	80	20			0	CNTs - for FTIR and SEM analyses
CSiO-07N	12/12/14	1.8E-07	27	SN20N	BN	900	20	700	6000	80	20			0	Small CNT spots delaminate due to static electricity from a Petri dish cover - Poor Ni adhesion?
CSiOc01N	12/12/14	1.8E-07	27	SN21	BN	900	20	700	6000	80	20			0	No CNTs - Poor Ni adhesion? CNT delamination by gas flow in reactor?

C. Sputter deposition with the "Plasmionique SPT330" system

Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle_Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
YBCO-A	12/09/17	7.9E-07	2	YBCO	3	Si	45	10	10		19		RF-2	200	0	76	2							2	18	M	0	OR	153	93	M			Coating and target cleaning - Turbo pump was stopped to allow that high pressure
											78		RF-2	200	0	54		180						2	18	M	0	OR	143	77	M	116		idem + Material coated inside chimney walls peels off: flakes fall down when shutter opens
YBCO-B	12/09/19	9.7E-07	2	YBCO	3	Si	45	10	10		19		RF-2	200	0	108	2		50	0	164	2		2	18	F	96	12		9.4	M			
											66		RF-2	200	1	90		125						2	18	F	96	12		4.8	M	252		
YBCO-C	12/09/20	5.4E-07	2	YBCO	3	Si	45	10	10		17		RF-2	200	1	89	2							5	45	F	90	12		8.4	M			Same pressure as YBCO-B but higher gas flow (2.5X)
											69		RF-2	200	1	78		125						5	45	F	90	12		6	M	210		
YBCO-D	12/09/20	2.0E-07	2	YBCO	3	Si	45	10	10		51		RF-2	200	1	52	2							5	45	F	97	42.5			M			
											94		RF-2	200	1	51		125						5	45	F	97	42		5.9	M	118		

Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
YBCO-E	12/09/24	2.9E-07	2	YBCO	3	Si	45	10	10		16		RF-2	200	1	48	2							2	18	M	0	OR	147	81	M			Same parameters as YBCO-A but different properties. Turbo pump was stopped to allow that high pressure
											83		RF-2	200	0	33		125						2	18	M	0	OR	142	80	M	47		Dektak shows a high surface roughness
Ti-14	12/11/16	5.9E-07	1	Ti	3	Si back	45	10	10		16		RF-1						100	0	250	3			45	M	92	17.6			M			
											18		RF-2	250	1	266	2	120	100	1	223	30			45		92	13.9						Material coated inside chimney walls peels off: flakes fall down when shutter opens
Ti-15	12/11/20	5.8E-07	1	Ti	3	Si	45	10	10		21		RF-1						100	0	236	10			45	M	76	5.1			M			
											31		RF-2	250	1	237	2	60				2			45		76	5.1				476		
																																		New 200sccm Ar mass flow installed and tested with David (replaces old 50sccm)

Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
Ti-16	12/11/21	3.5E-07	1	Ti	3	Si	45	10	10		25		RF-1						100	1	234	10			150	M	76	15.1	15.4		M			
											35		RF-2	250	1	237	2	60				2			150		76	15.1	15.4			361		
Ti-17	12/11/22	5.4E-07	1	Ti	3	Si	45	10	10		21		RF-1						100	0	238	10			141	M	0	5			M			
											33		RF-2	250	1	237	2	60				2			140		0	5.1	5.5			471		
Ti-18	12/11/22	2.9E-07	1	Ti	3	Si	45	10	10		36		RF-1						100	0	230	10			45	M	75	5	5.04		M			
											45		RF-2	400	1	288	2	60				2			45		75	5	5.04			839		
Plasma Etch	12/11/28	3.0E-07		Substrates		SiN-0F Ti	45	None	10		15		RF-1						100	0	260	0			45	M	87	10	10.7	7.9	M			
Plasma Etch				below							30		RF-1						100	0	229	20			45	M	87	10.2	10.7	8	M			
Plasma Etch											91		RF-1						100	2	212	80			45	M	87	10.2	11.7	7.9	M			
Si-00_pe				Si-00																												237		
Ti-16_pe				Ti-16																												39		
Ti-17_pe				Ti-17																												21		
Ti-18_pe				Ti-18																												22		
SiN-0F_pe				SiN-0F																												242		
YBCO-F	12/12/07	7.4E-08	2	YBCO	3	Si	45	10	10		19		RF-1						100	1	233	4		5	45	M	85	10.3		7.6	M			
											20		RF-2	200	0	112	2							5	45	M	74	5		6.4	M			
											70		RF-2	200	0	102		120						5	45	M	74	5		7.1	M	250		

Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
YBCO-G	12/12/10	1.3E-07	2	YBCO	3	Si	45	10	10		17		RF-1						100	1	224	2		5	45	M	85	10.2		8.6	M			
											16		RF-2	200	0	101	2						5	45	M	74	4.8		6.9	M			
											70		RF-2	200	1	89		120					5	45	M	74	5		7.7	M	198		Surface roughness of the coating is higher at the outer 1cm edge of the 4" wafer
YBCO-H	12/12/11	5.4E-08	2	YBCO	3	Si	45	10	10		21		RF-1						100	0	217	2		5	45	M	84	10	10.5	7.9	M			Pfeiffer zero verified, pins3-4=1v ok
											20		RF-2	200	1	78	2						5	45	M	84	10.1		8.9	M			
											77		RF-2	200	0	66		120					5	45	M	84	10		8.9	M	138		High Surface roughness of the coating
YBCO-I	12/12/11	1.2E-07	2	YBCO	3	Si	45	10	10		59		RF-1						100	1	207	2		5	45	M	85	10.1		7.7	M			
											58		RF-2	200	1	66	2						5	45	M	92	15.1		9.9	M			
											91		RF-2	200	0	64		113					5	45	M	93	15.1		5.6	M	137		Deposition stopped due to observed power instabilities- Coating on chimney inside walls delaminates during this deposition - High Surface

Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																																		roughness of the coating
YBCO -J	12/12/12	8.0E -08	2	YBCO	3	Si	45	10	10		19		RF -1						100	1	200	2		5	45	M	84	10		7.5	M			
											19		RF -2	200	0	78	4						5	45	M	58	2.5	3.12	3.7	M			
											30		RF -2	200	0	108		41					5	45	M	58	2.5	3.1	5.6	M			Deposition ok - I left after 30 min
											39		RF -2	93	93	1		48					5	45	M	58	2.5	3.1	5.6	M	70		Deposition stopped (target bias nulled and Reflected/Fw power rose/dropped to 93W, Plasma off) for no obvious reason after a total of 41min. I noticed this 48min later and technically shut off the RF2 generator
	12/12/21																																	RF1&2 not working but DC-1 ok! DEBUG.

Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																																			Manual Matching from high values works awhile. Needs starting pressure 20-30mTorr
Bolo9 Ti	13/01/07	2.3E-07		Bolo9			45	None	10		15		RF-1						80	0	14	1			65	M	95	29.2		13	M			
											16		RF-1						100	0	14	20			45	M	87	10	10.5	8	M			8nm Ti and 48nm Si etched
			1	Ti	3	Bolo9	45	10	10		25		RF-2	400	1	294	5					1			45	M	77	5		5.8	M			
											76		RF-2	400	2	281		179							45		76	5	5.32	5.3	M	2508		2508nm =48nm (Si etched) + 2460nm measured with dektak on the Si holder
Bolo2 Ti & Bolo5 Ti	13/01/11	4.7E-08		Bolo2 & Bolo5							16		RF-2	200	0	124	5								65	M	76	7.3		5.9	M			Target cleaning, At 5mTorr Bias drops from 132V to 0V after 2min so I increase the pressure to restart the plasma
							45	None	10		15		RF-1						100	1	20	1			65	M	95	30.6		13	M			

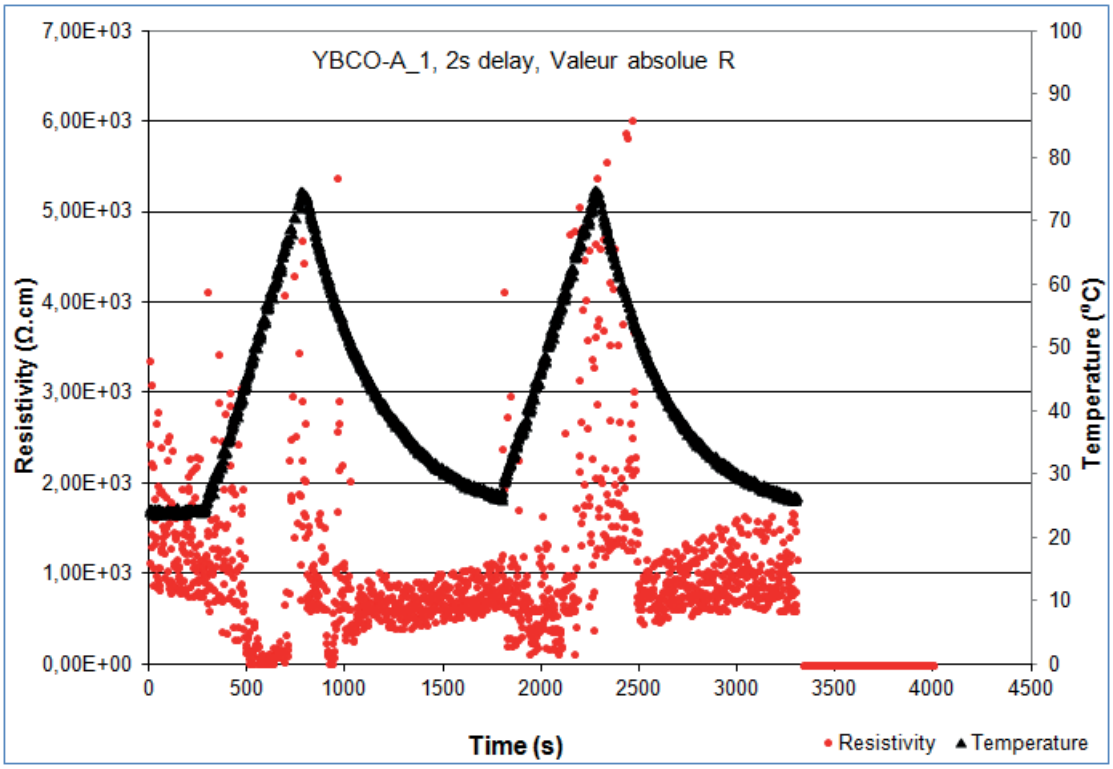
Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
											56		RF -1						100	1	8	83			45	M	87	10	10.2	7.1	M			
			1	Ti	3	Bolo 9	45	10	10		57		RF -2	400	1	297	1					0			45	M	76	5.1		5.7	M			
											98		RF -2	400	1	270		**250**							45		76	5	5.1	5.1	M	3500		Reflected/Fw power rises/drops to 96W at 2h45 for 26min - Plasma was probably off (opaque windows). I restarted the deposition, adding 26min to planned time; 3623nm =18nm Ti etched + 3605nm dektak, 3891nm =197nm Si etched +3694nm dektak
	13/01/14																																	New YBCO target received is blacker than the previous and seems to be of better

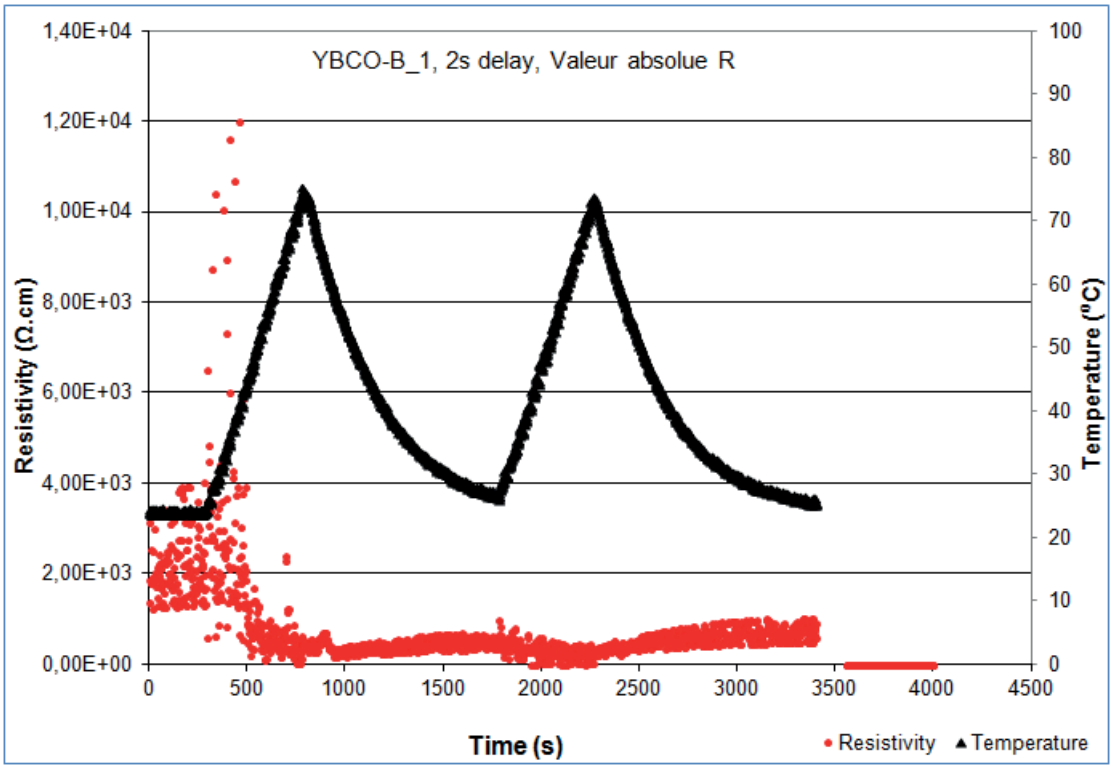
Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																																		quality. I replace the previous one (which reveals to be fissured) with this on Gun 2
Si-01_pe	13/01/15	3.0E-07		Si		Si	45	None	10		16		RF-1						100	0	24	0			45	M	87	9.9		7.1	M			
Plasma Etch											60		RF-1						100	1	6	100			45	M	87	9.8	10.2	7	M	0!		Dektak : less than 9nm Si etched with that low voltage. Recent Ti depositions probably created a thin film short (Resistance on substrate holder connector is 1.17k_ohms)
Time tests	13/01/16	2.0E-07		None		Si	45	None	10		16							a							0									To verify the correspondence between Stopwatch Time and Log-File Time
																		0							50	M	0	1.8	1.66	1.8	M			
																		20							50	M	0	1.8	1.6	1.	M			

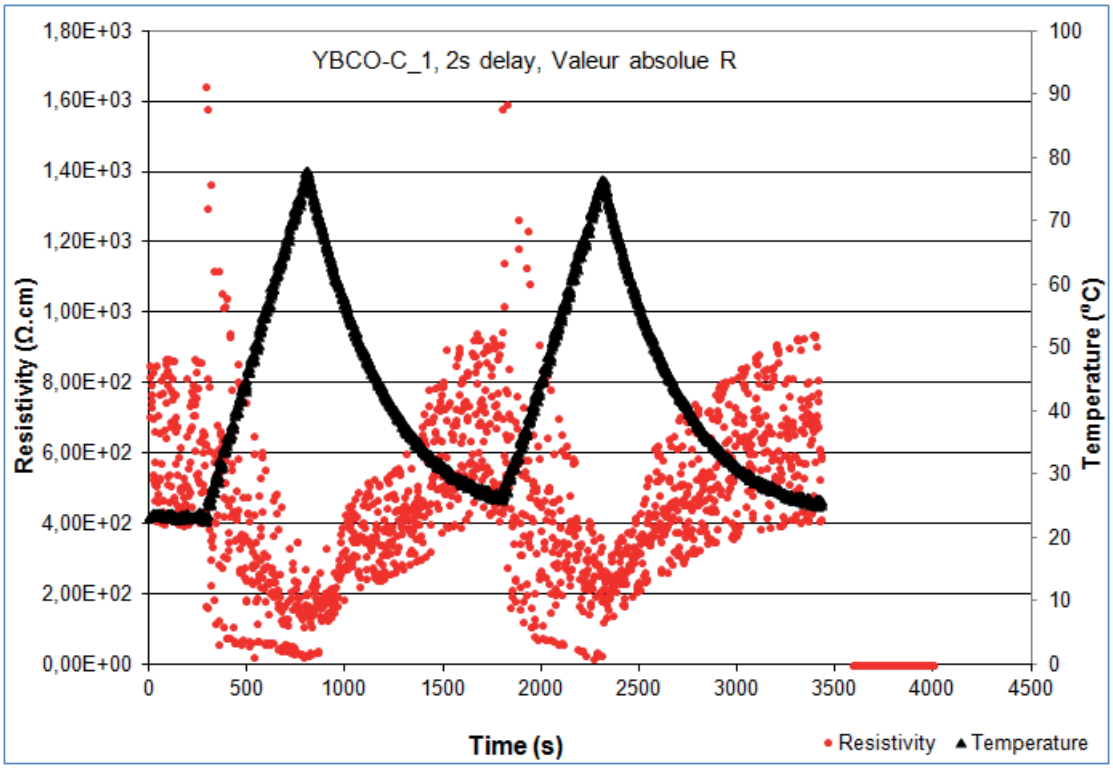
Sample name	Date	Base Pressure - Varian - Ion Gauge (Torr) Pres1	Gun Number	Target Material	Gun/Target diameter (")	Substrate	Substrate Position (mm)	Target→Substrate Distance (cm)	Substrate Rotation Velocity (rpm)	Temperature Setpoint Value SV (°C)	Temperature Probe Value PV from TC1 (°C)	Real Sample Temperature (°C)	Mode-Match : RF-2, DC-1	Sputtering FW Power (W)	Reflected Power (W)	Cathode Voltage (V)	Presputtering Time (min)	Sputtering Time (min)	Bias FW Power - sample (W)	Bias Reflected Power (W)	Bias Voltage - sample (V)	Bias Duration (min)	N2 flow (sccm)	O2 flow (sccm)	Ar flow (sccm)	Pressure Control: Manual, Throttle, Flow	Throttle Valve (% Closed)	Deposition Total Pressure - MKS - Baratron (mTorr)	Deposition Total Pressure - Pfeiffer - CMR (mTorr)	Deposition Total Pressure - Varian - Pres1 (mTorr)	Program: Manual, Automatic	Thickness (nm) Dektak	Thickness (nm) Ellipsometry	Comments
																		0											6	8				
																		z							0									
Si-02_pe	13/01/16	1.3E-05		Si		Si	45	None	10		18		RF-1						100	3	239	0			45	M	87	10.3		8.4	M			Resistance on substrate holder connector is 0,911k_ohms. Cleaning of substrate holder (IPA, Acetone, dirt suction with a vacuum, N2 blow, wipes) yields 620k_ohms
Plasma Etch											33		RF-1						100	1	216	25			45	M	87	9.9		8.1	M	0!		

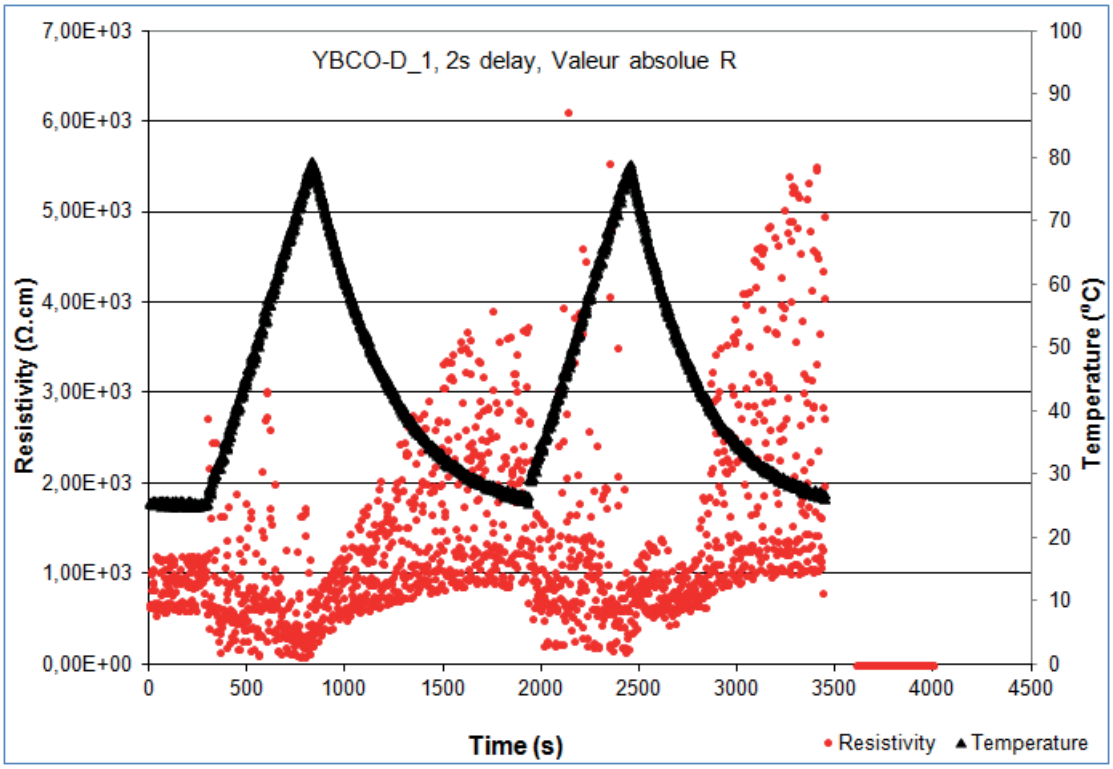
Annex 2: Resistivity/Temperature data for YBCO samples

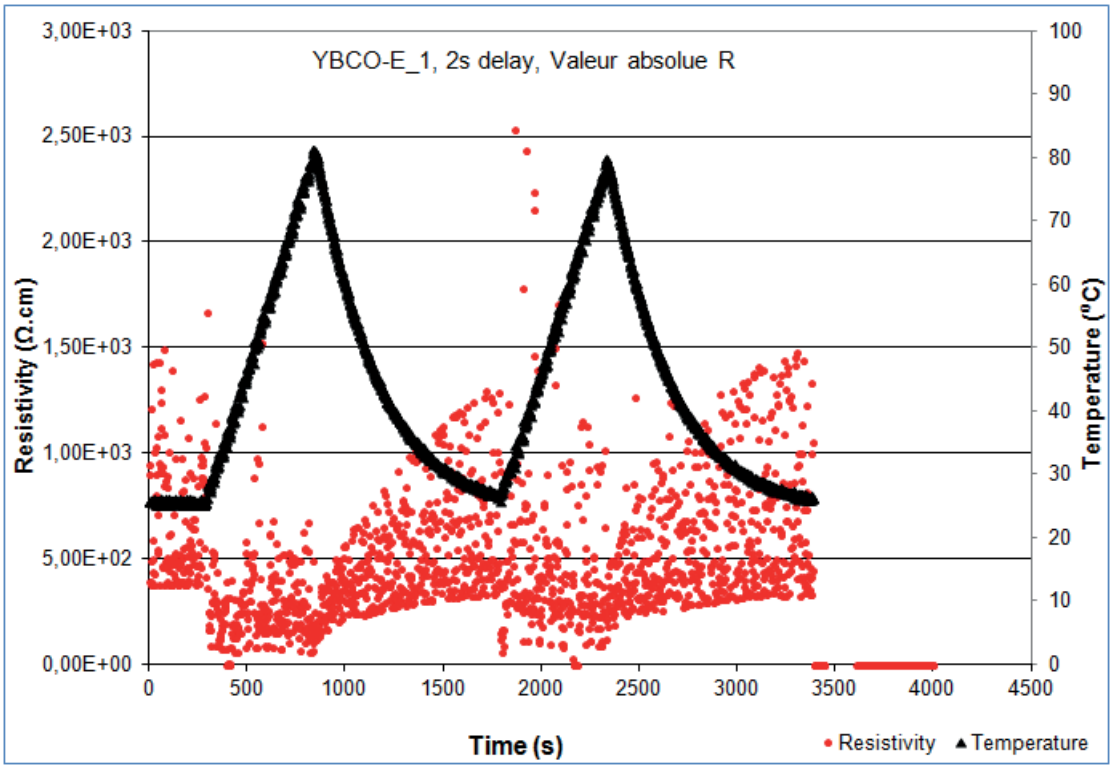
Measurements performed using the Signatone Pro-4 Manual Four Point Probe

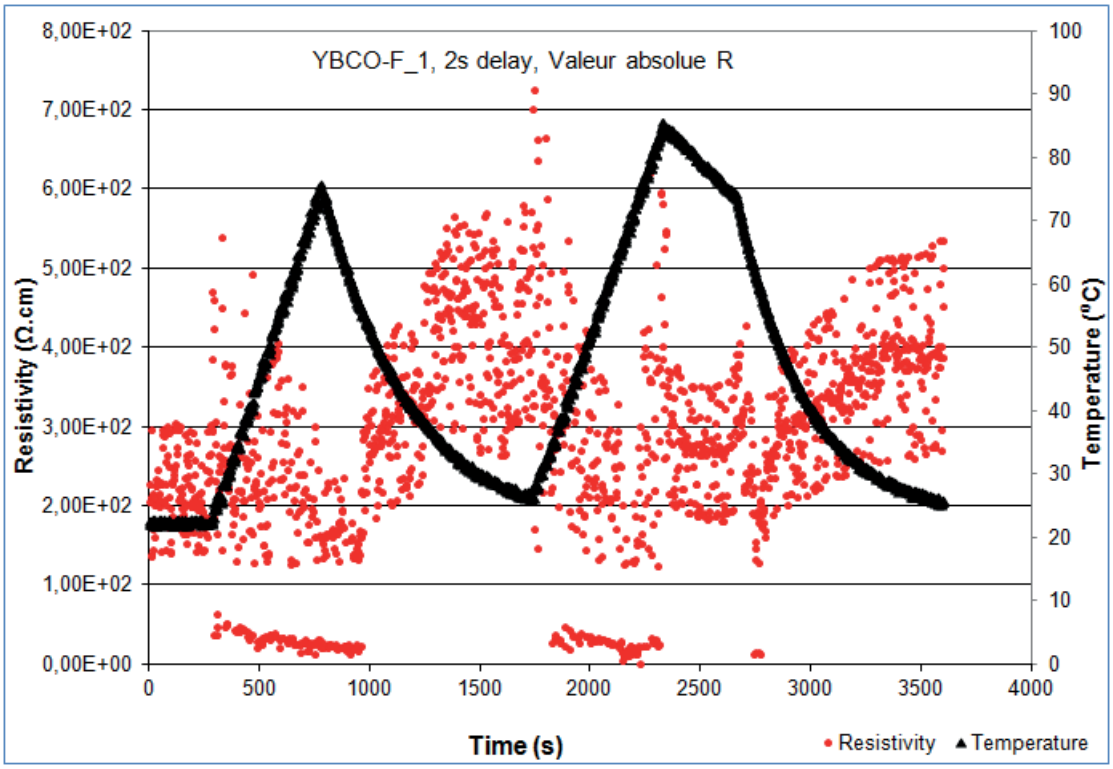


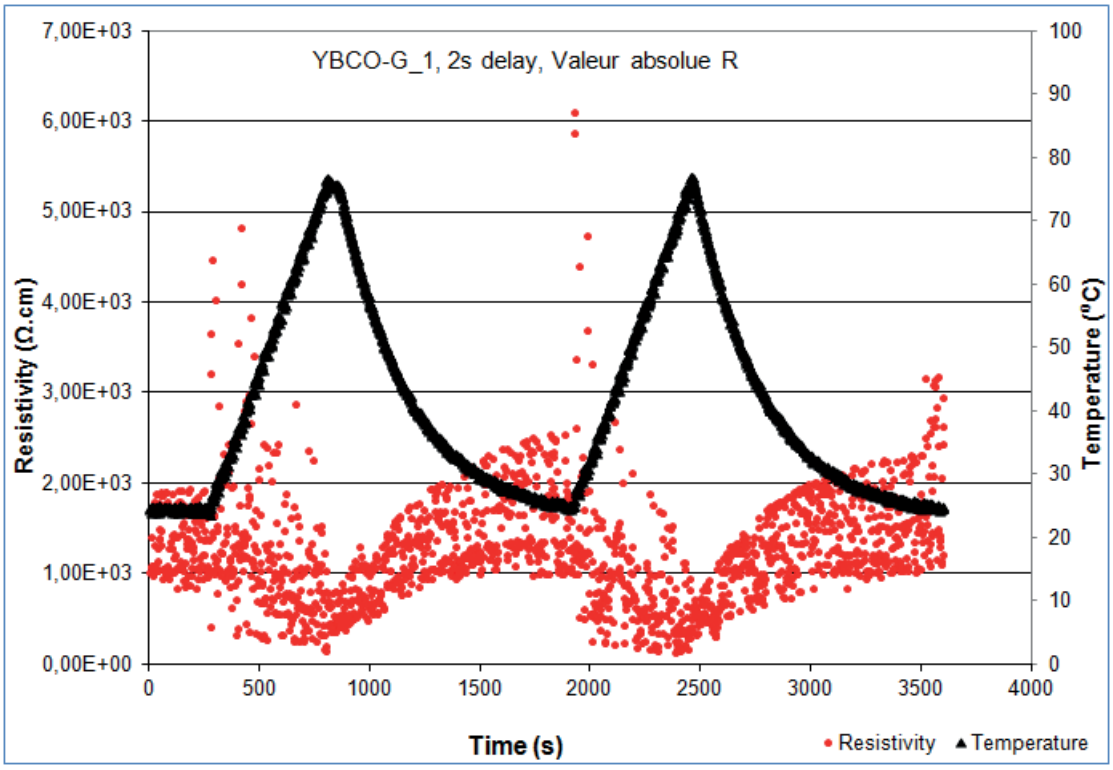


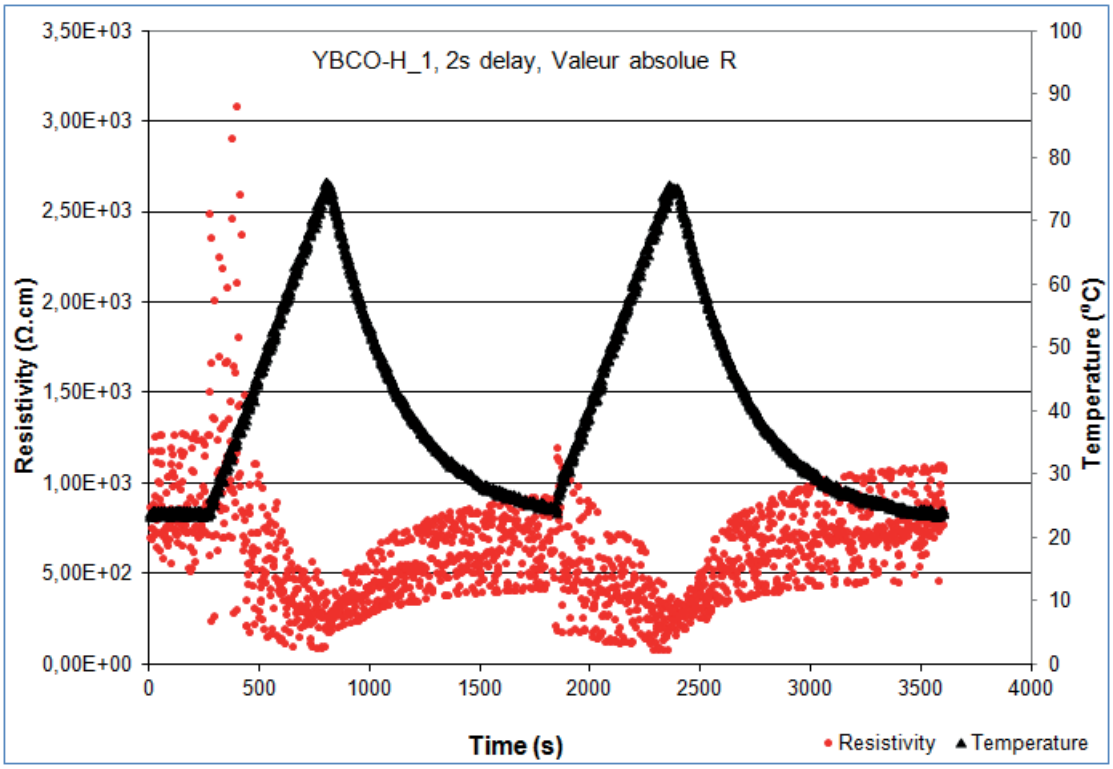

YBCO-C_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature

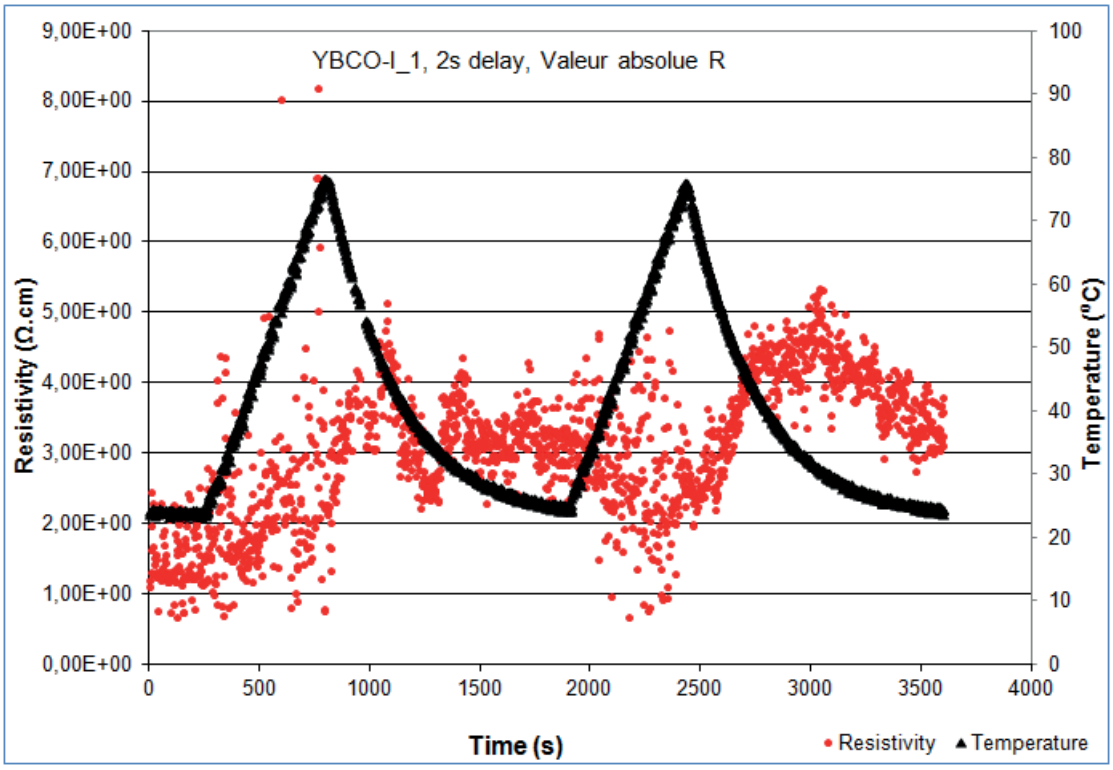

YBCO-D_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature

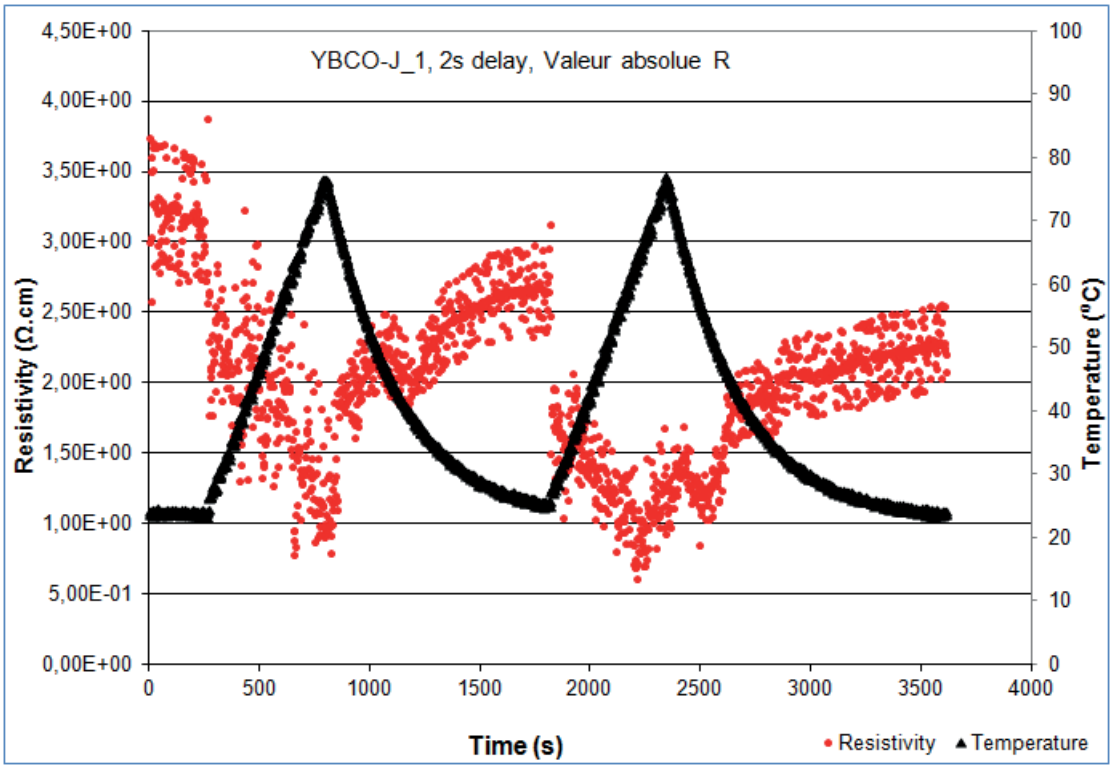

YBCO-E_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature


YBCO-F_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature


YBCO-G_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature
7,00E+03
6,00E+03
5,00E+03
4,00E+03
3,00E+03
2,00E+03
1,00E+03
0,00E+00
100
90
80
70
60
50
40
30
20
10
0
0
500
1000
1500
2000
2500
3000
3500
4000


YBCO-H_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature
3,50E+03
3,00E+03
2,50E+03
2,00E+03
1,50E+03
1,00E+03
5,00E+02
0,00E+00
100
90
80
70
60
50
40
30
20
10
0
0
500
1000
1500
2000
2500
3000
3500
4000


YBCO-I_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature
9,00E+00
8,00E+00
7,00E+00
6,00E+00
5,00E+00
4,00E+00
3,00E+00
2,00E+00
1,00E+00
0,00E+00
100
90
80
70
60
50
40
30
20
10
0
0
500
1000
1500
2000
2500
3000
3500
4000


YBCO-J_1, 2s delay, Valeur absolue R
Resistivity (Ω.cm)
Temperature (°C)
Time (s)
Resistivity
Temperature
4,50E+00
4,00E+00
3,50E+00
3,00E+00
2,50E+00
2,00E+00
1,50E+00
1,00E+00
5,00E-01
0,00E+00
100
90
80
70
60
50
40
30
20
10
0
0
500
1000
1500
2000
2500
3000
3500
4000